

# Diamond film nano-abrasives obtained by anodic arc

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Thermo ionic Vacuum Arc plasma (TVA) was employed to synthesize H-free DLC films containing nanocrystalline grains embedded in an amorphous structure, at high deposition rates of 1.5  $\mu\text{m}/\text{h}$ . We have previously demonstrated that the grains observed in the film are diamond crystallites. The results presented in the current paper show a direct correlation of the size of the grains with distance from the anode. It was found that larger grains of the order of 100-200 nm but fewer are formed closer to the anode, whereas very smooth films with 1-2 nm roughness were obtained at 30 cm away from the anode. The control of DLC film nanoscale roughness opens the way for the fabrication of diamond devices and nano-abrasives.

(Received October 19, 2009; accepted October 29, 2009)

*Keywords:* TVA plasma, Diamond films, Anodic arc, Nano-abrasive

## 1. Introduction

Polycrystalline diamond films have large grain sizes, resulting in very high surface roughness of the order of micrometers. There are applications where finer roughness is necessary such as: surface acoustic wave (SAW) devices, MEMS, optical components, nano-abrasives etc. Finer surfaces with roughness at the nanoscale are therefore being investigated also. There are still little advances in this field, as increased diamond nucleation at this scale needs to be based on other methods than surface treatment with diamond powder in an ultrasonic bath. Substrate negative bias in hot-filament chemical vapour deposition (HFCVD) was reported as a potential candidate for obtaining diamond films with nanometric roughness [1]. The disadvantage of this technique for industrial applications is the fact that the nanoscale roughness is obtained at the backside of the diamond film (at the film substrate interface), further processing involving chemical etching of the substrate being also needed. In this paper, an original method for the controlled synthesis of DLC films containing nanoscaled diamond grains is presented. The method is known as Thermo ionic Vacuum Arc (TVA) and has particular features which make a special case in the synthesis of films with desired properties.

## 2. Experimental details

The TVA method is based on the ignition of an arc plasma in the vapours of the anode, here graphite. Full details of the TVA system are presented elsewhere [2]. A particular feature of the TVA method is the use of a specially designed electron gun which has a double role:

heating of the anode material (consisting of a graphite rod) and ionization of the neutral Carbon atoms created during evaporation. The precursors of the film are: neutral atoms of carbon and a small percentage of very energetic carbon ions. As the deposition system works under high vacuum of about  $10^{-6}$ Torr and no buffer gas is needed, the films deposited are hydrogen-free and very pure. This precursor configuration (in terms of type, relative density and energy) is unique and therefore the films obtained also have particular features. Typically, the Carbon ion energies obtained in TVA are of a few hundreds eV and can achieve 1000eV [3]. Such high values of the ion energy are due to the fact that the ions generated in the plasma travel towards the chamber walls without collisions. They are repelled by the plasma and their energy is given by the potential difference between the plasma potential ( $V_p$ ) and the potential of the chamber walls (which are grounded):  $E_{\text{ion}} = e(V_p - V_{\text{walls}}) = eV_p$ . As  $V_p$  is proportional to the applied voltage  $V_{\text{applied}}$ , a variation of  $V_{\text{applied}}$  results in a variation of the ion energy in the same direction.

The films studied in this work were deposited using different values of the arc voltage in the range 400 – 1000 V and the distance between the anode and the substrate was varied between 4 and 22cm.

AFM images of the DLC films obtained by TVA were acquired using a scan rate of 1Hz and 512 data points were collected along each of the 512 scan lines. The force between the tip and sample was 10.8nN.

As already known, exact measurement of the  $sp^3$  content is not completely established by usual techniques like XPS or Raman. This is the reason why film hardness was chosen to qualitatively determine the relative  $sp^3$  content of our DLC films.

The hardness of our films was measured using a CSM Nano Hardness Tester (NTH).

### 3. Results

As previously found, the TVA plasma ignited in Carbon vapours produces DLC with crystals of  $sp^3$  diamond bonding embedded in a graphitic amorphous structure [5]. The current paper reports on further studies of the influence of plasma operating parameters on the morphology of the films.

The size of diamond crystallites was found to depend on the distance to the anode ( $D_a$ ). AFM images of films deposited at 22, 16, 10 and 4cm are shown in Figures 1–4. As can be observed in these images, the films closer to the anode present round features of over 100nm diameter.

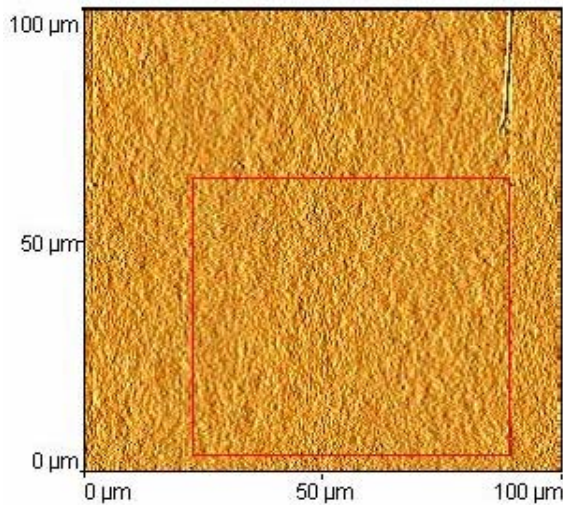


Fig. 1. AFM images of DLC films deposited at 22 cm.

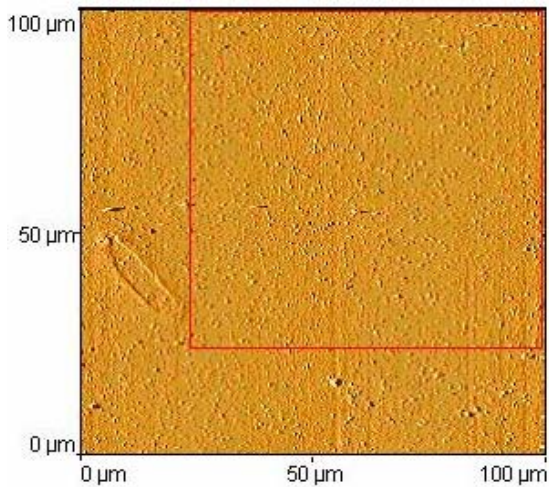


Fig. 2. AFM images of DLC films deposited at 16 cm.

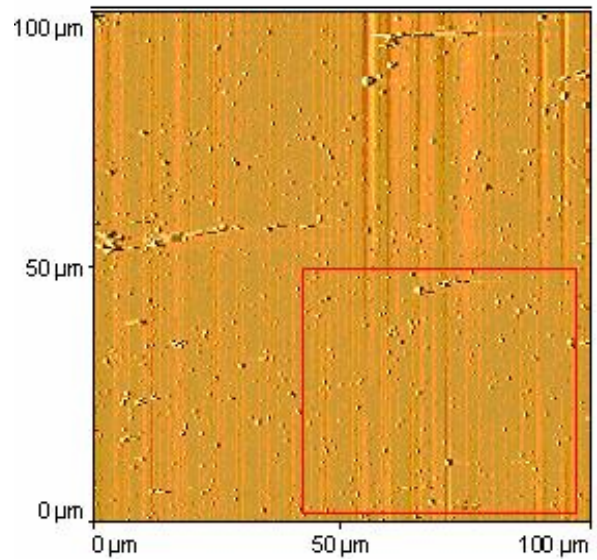


Fig. 3. AFM images of DLC films deposited at 10 cm.

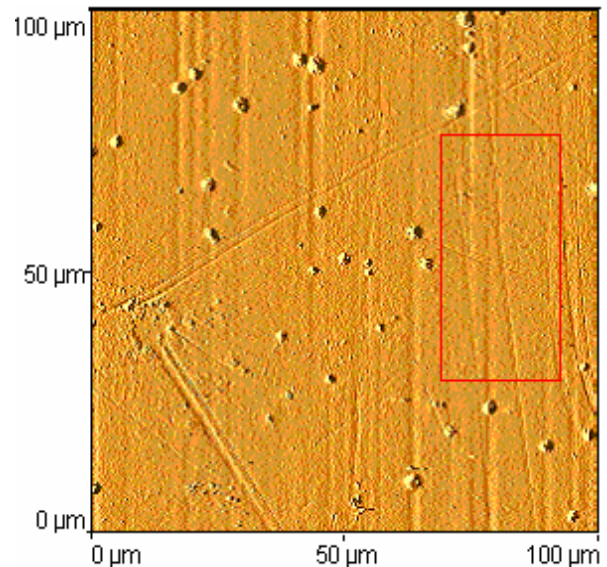


Fig. 4. AFM images of DLC films deposited at 4 cm.

The C ion energy is a key factor in obtaining  $sp^3$  bonds. It is known that the ion energy is inversely proportional to the hardness for ion energies in the (200–500)eV range [4]. As can be observed, the hardness of all films studied ranged between 7.3 to 13.5 GPa and varied monotonically with the applied voltage. As the hardness is directly proportional to the  $sp^3$  fraction, these results suggest that a higher  $sp^3$  content was obtained at lower applied voltage.

The hardness of all films studied was between 7.3 to 13.5GPa and the applied voltage was varied between 1000 and 400 V.

A very low roughness of 2nm was obtained on the DLC films deposited by the TVA method without using additional filters.

The deposition rate was about 1.5 $\mu$ m/h, which makes TVA an industrially competitive technology.

#### 4. Conclusions

This is the first time the Thermo ionic Vacuum Arc (TVA) plasma source was used to synthesize DLC films with controlled nanoscale roughness and sp<sup>3</sup> content.

In conclusion, the DLC films obtained by carbon TVA plasma are amorphous, very smooth, hard and have nanoscale features of controllable size.

The quality of the films, the high deposition rate and the controllability of the deposition parameters make the TVA plasma a special case in the synthesis of films with desired properties.

The results are important because such films can be used in nanoscale diamond devices and nanometric abrasives.

#### Acknowledgements

The authors wish to thank the Ministry of Education, Research and Youth (MECT) – Bucharest Romania for financial support.

#### References

- [1] S.-M. Huang, H.-C. Hsu, M.-S. You, F. C.-N. Hong, *DRM* **15**, 22 (2006).
- [2] C. Surdu-Bob, I. Mustata, C. Iacob, *J. Optoelectron. Adv. Mater.* **9**(9), 2932 (2007).
- [3] C. C. Surdu-Bob, M. Badulescu, C. Iacob, C. Porosnicu, C. P. Lungu, *Journal of Physics: Conference Series*, IWSSPP, in print.
- [4] J. Robertson, *Mater. Sci. Eng. R* **37**, 129 (2001).
- [5] C. Surdu-Bob, R. Vladoiu, M. Badulescu, G. Musa, *Diamond and Related Materials* **17**, 1625 (2008).

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